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COMPARISON OF SELECTED PROPERTIES OF NATURAL AGED WOOD AND CONTEMPORARY TIMBER OF *PINUS SYLVESTRIS* L. INVESTIGATED USING STANDARD METHODS AND MEASURING OF TRANSITION SPEED OF ULTRASOUNDS ALONG THE FIBRE

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ABSTRACT

Scots pine wood (*Pinus sylvestris* L.) is the most common wood material used in historical buildings in many parts of Central and Eastern Europe. Experiments were conducted natural aged wood (263 – 459 years old), extracted from construction elements of four historical buildings (from seven construction elements), and contemporary wood extracted from 5 construction elements. A strong relationship was observed between density and static bending strength (MOR) of natural aged wood (R^2 = 0.5599), and also of contemporary timber (R^2 = 0.7863). Antique wood compared to contemporary wood with the same average moisture content and density is characterized by significantly lower modules (static and dynamic), the speed of ultrasonic waves transitions, and bending strength. Differences in these properties increase with increasing wood density.

KEYWORDS: *Pinus sylvestris*, natural aged wood, physical properties, mechanical properties.

INTRODUCTION

Mechanical properties of natural aged wood have been of great interest among researchers for many years. A great contribution into research on natural aged wood was made between 1950-1955 by Kohara (1952, 1954, 1955, 1956), who tested *Chamaecyparis obtusa* Endl. and *Zelkowa serrata* Thunb. wood taken from very old constructions of Japanese temples (Obataya 2007). Some constructions were even 1300 years old. In the research of Yokoyma et al. (2009), experiments for static bending conducted on *Ch. obtusa* wood samples cut from historical constructions proved that the technical properties of the wood were still quite good as compared to contemporary wood.

Scots pine (*Pinus sylvestris* L.) is a dominant species of wood in the middle of Central-Eastern Europe. In some regions it has been the main building material (e.g Mazovia and Warmia regions in Poland). However, natural aged coniferous softwood from Europe, including Scots pine wood (Kräniz et al. 2010) and spruce wood (Schulz et al. 1984, Kräniz et al. 2010, Thaler and Miha 2013), is a relatively rare subject of research on mechanical qualities. Also, European natural aged coniferous wood used in such research is usually much younger – around 100÷200 years old (Rug and Linke 2013, Thaler and Miha 2013, Kräniz et al. 2010, Yorur and Yumrutas 2014) – than aged wood of *Ch. obtusa* examined in Japan (Yokoyma et al. 2009). The age of *Ch. obtusa* samples used in that research ranged from 500 to over 1600 years.

Among laboratory tests on mechanical qualities of aged wood from historical constructions, the most common topic is definitely measuring compressive strength parallel to grain (Deppe and Ruhl 1993, Obataya 2007, Witomski et al. 2014, Sonderegger et al.2015). Results of research on other qualities of European aged wood from historical constructions are rather not as common. They include research on shear strength of aged Scots pine heartwood (Yorur and Yumrutas 2014, Krajewski et al. 2016).

Special reviews in this regard are the reviews on the mechanical properties of antique wood (Cavalli et al. 2016, Kränitz et al. 2016). These works, however, focus on other species of wood (properties of Scots pine wood are presented at random mainly in terms of compression strength).

This insufficient research raises the question. How, in the case of naturally aged wood of Scots pine wood, do such properties as wood static bending strength (MOR) and Young modules (dynamic and static MOE), as compared to contemporary timber? This is an important issue, among others, in assessing the safety of the wooden constructions and the decision on the need and scope of conservation work undertaken.

The possibility of using measurements of transition speed of ultrasounds along the fibre in order to estimate wood static bending strength (MOR) seems an interesting option. For this reason, the correlation between static bending strength (MOR) and density of wood, and the correlation between transition speed of ultrasounds along the fibre of wood and density of wood were studied.

MATERIALS AND METHODS

The authors of the present research used natural aged heartwood of Scots pine (*Pinus sylvestris* L.) free from fungal decay or insect attack. The tested wood samples were taken from 4 historical constructions form Poland (Mazovian district and Warmian-Masurian district) dating from 16^{th}-18^{th} century. Samples used in tests were sized 20 x 20 x 300 mm and extracted from 7 constructional elements: (A1) wall beam from the church in Puszcza Mariańska (dating from 1755) – 8 samples, (A2) wall beam from the church in Puszcza Mariańska (1755) – 17 samples, (A3) wall beam from the church in Puszcza Mariańska (1755) – 13 samples, (A4) element of rafter from the monastery in Święta Lipka (German: Heilige Linde, 1703) – 4 samples, (A5) element of rafter from the castle in Lidzbark Warmiński (German: Heilsberg, 1559) – 17 samples, (A6) element of rafter from the church in Cegłów (before 1629) – 10 samples, (A7) element of rafter from the church in Cegłów (before 1629) – 30 samples.

The wood collected for research was dated based on the analysis of the documentation of the objects from which it came. The authenticity of the elements was confirmed on the basis of their appearance and location in the constructions and presence of the original carpentry marks.

The age given is the number of years of use in the construction (the actual age of antique wood is higher). Natural aged wood was represented by a total number of 99 samples taken from constructions aged 263 – 459 years. The wood from the castle in Lidzbark Warmiński was taken from trees cut down at the age of 130 – 140 years. The wood from all the other constructions was taken from trees cut down at the age of 100 – 120 years.

The contemporary timber was taken from 5 Scots pine heartwood timber from tress at the age of around 80 – 100 years. 75 samples of dried and seasoned timber were used for comparative tests (F1 - 5 samples, F2 - 12 samples, F3 - 18 samples, F4 - 20 samples and F5 - 20 samples). Density of natural aged wood and fresh cut and dried and seasoned timber of each sample was calculated using stereometrical method according to standard method ISO 12061-2: 2014.

Moisture content (MC) of the wood was determined using oven-dry method according to ISO 13061-1: 2014. The transition speed of ultrasounds along fibre and dynamic modulus of elasticity (MOEd) were determined using an original ultrasonographic method with the use of material tester UMT-1, connected to specialistic computer software. The tests were conducted using two cylindrical heads of 40 mm diameter: receiving head and transmitting head producing ultrasounds of frequency of 40 kHz. The tester worked in impulse mode (transmission 12 Hz) with 40 dB gain and at the electrical voltage of 60 V, using policrylate gel as coupling substance. The pattern delay time was t0 = 8.9 μs.

After running ultrasound waves parallel to the grain, the time of the main echo was read and the results were used to calculate the speed of longitudinal waves parallel to the grain:

$$c_{\parallel} = \frac{L}{t} \tag{1}$$

where: c - speed of the longitudinal waves parallel to the grain ($m \cdot s^{-1}$)
L - sample length [m] (assuming that L >> λ)
$t = t_1 - t_0$ - real time of the passing through of the longitudinal wave (s)
t_1 - time of the passing through of the wave read from the computer screen (s)
t_0 - lag time (s).

On the basis of speed the elasticity, modulus parallel to the grain was calculated using the formula below:

$$MOE_d = c_{\parallel}^2 \cdot d \cdot \frac{(1+\mu_o)\cdot(1-2\cdot\mu_o)}{(1-\mu_o)} \tag{2}$$

where: MOE_d - dynamic modulus of elasticity parallel to the grain (GPa)
μ_o - reduced Poisson's ratio for wood (μ_o = 0.3) (-)
d - density of wood of a known moisture content ($kg \cdot m^{-3}$).

Average values of transition speed of ultrasounds through and average the dynamic elasticity modulus both aged wood and fresh cut timber were then calculated. Statistical significance of the difference between those values was evaluated using Student test. The relationship between transition speed of ultrasounds along fibre in natural aged wood and contemporary timber and wood density was determined and coefficients of determination (R^2) were calculated.

Static bending strength of wood (MOR) and static modulus of elasticity of wood (MOEs) were tested for each sample according to standard method ISO 13061-3:2014 and ISO 13061-4:2014. To establish those values, a universal testing machine INSTRON (model 3369) was used. The machine is connected to computer program INSTRON Series IX/s Automated Materials Tester Version 8.32.00, allowing the analysis of the results.

Average values of MOR, static MOE and dynamic MOE for both aged wood and contemporary timber were calculated. Also, coefficients of variation were calculated for each of those features for every set of samples from each single construction element, as well as for the whole sets of samples of both natural aged wood and contemporary timber. It was verified whether the obtained groups of results for both aged wood and fresh cut timber exhibit normal distribution. Next, statistical significance of differences between average values of MOR, static MOE and dynamic MOE was evaluated. To determine statistical significance of the differences between those properties of aged wood and contemporary timber, Student test was used, as those values exhibited normal distribution. The relationships between MOR, static MOE and dynamic MOE, and wood density, were determined. Also, coefficients of determination (R^2) were calculated.

RESULTS AND DISCUSSION

The results of measurements and calculations are shown in Tab. 1 in the form of numerical data. The average values of wood properties and their coefficients of variation for each sample of natural aged wood and contemporary timber displayed some differences. The results of statistical verification of significance of the differences between the average values of moisture content, density, transition speed of ultrasounds along fibre dynamic MOE, static MOE, and bending strengths (MOR) for both natural aged wood and contemporary timber are shown in this table too.

Tab. 1: Selected physical and mechanical properties of natural aged heartwood and contemporary heartwood of scots pine (Pinus sylvestris L.).

No of elements	**Number of samples**	**MC (%)**	**Density of wood ($kg·m^{-3}$)**	**Transition speed of ultrasounds‖ ($m·s^{-1}$)**	**MOEd (MPa)**	**MOEs (MPa)**	**MOR (MPa)**
Coefficient of variation (in brackets) *v* (%)							
A1	8	8.9 (3.1)	467 (3.6)	5362 (2.5)	9687 (6.2)	8793 (7.4)	73.9 (15.6)
A2	17	9.1 (2.4)	549 (4.1)	5449 (6.1)	11808 (14.6)	10409(13.3)	89.4 (15.6)
A3	13	8.8 (3.2)	418 (5.5)	5019 (5.5)	7616 (12.6)	7116 (12.0)	63.9 (8.9)
A4	4	8.9 (3.0)	452 (2.1)	5723 (1.1)	10671 (4.1)	9918 (6.7)	84.5 (3.3)
A5	17	8.7 (2.6)	365 (2.1)	5107 (1.6)	6853 (3.4)	6498 (3.8)	62.4 (7.9)
A6	30	9.0 (4.5)	459 (5.2)	5256 (6.9)	9203 (17.1)	8437(13.8)	81.8 (11.2)
A7	10	8.9 (3.8)	389 (6.5)	5026 (3.0)	7108 (10.8)	6678 (12.0)	56.9 (9.3)
Average for natural aged wood		**8.9 (5.2)**	**446 (14.0)**	**5237 (6.1)**	**8925 (23.4)**	**8180 (20.5)**	**74.4 (19.4)**
F1	5	8.9 (4.6)	531 (7.1)	5996 (2.0)	14216 (10.8)	12068(11.4)	111.9 (17.0)
F2	18	9.0 (2.4)	459 (11.2)	5777 (4.0)	11466 (18.0)	9955 (18.6)	91.0 (20.8)
F3	12	9.2 (3.9)	536 (3.4)	5897 (4.1)	13877 (8.8)	12141 (7.2)	108.5 (14.6)
F4	20	8.9 (2.6)	440 (10.1)	5343 (2.1)	9389 (15.6)	8255 (15.3)	83.4 (16.4)
F5	20	9.0 (2.8)	437 (7.1)	5460 (4.5)	9712 (13.3)	8517 (11.9)	84.4 (10.7)

Average for natural aged wood	**9.0 (6.3)**	**465 (11.8)**	**5610 (5.8)**	**11013 (21.4)**	**9609 (20.7)**	**91.4 (19.7)**
Statistical verification						
Differences between natural aged wood and contemporary wood	not significant	not significant	significant	significant	significant	significant

The antique wood, originating from various elements and objects, was characterized by considerable density variability from 365 kg·m^{-3} (element of rafter from the castle in Lidzbark Warmiński) to 549 kg·m^{-3} (wall beam from the church in Puszcza Mariańska). The coefficient of variation of this wood characteristic was 14%. Contemporary construction elements for research have been selected taking into account the similarity of the annual rings (width and arrangement) and density to the elements of historic wood.

The difference between the average density of natural aged wood and the average density of fresh cut timber proved to be statistically insignificant in the case of the tested samples at the assumed probability level. This fact allowed to compare all the other features of the wood. The tested wood did not show differences in moisture content. The differences between transition speed of ultrasounds along fibre, dynamic MOE, static MOE and MOR for both natural aged wood and contemporary timber were in all the cases statistically significant (Tab. 1). Thus, they cannot be explained by the difference in wood moisture content (MC) and density.

The relationship between density and static bending strength (MOR) of wood is shown in Fig. 1, separately for the tested collections of samples of natural aged wood and contemporary timber.

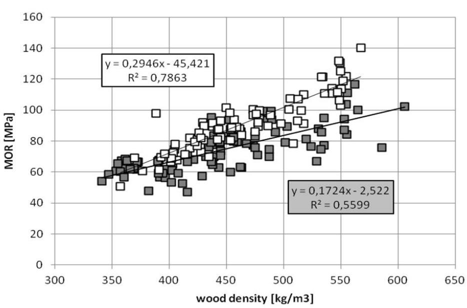

Fig. 1: The relationship between density and static bending strength (MOR) of natural aged wood (grey points) and contemporary timber (white points).

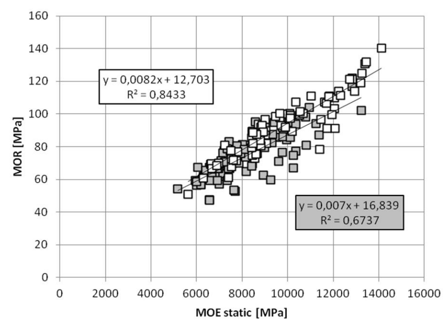

Fig. 2: The relationship between static MOE and static bending strength (MOR) of natural aged wood (grey points) and contemporary timber (white points).

Next, the relationship between MOR and static MOE is presented. The relationship between wood static bending strength (MOR) and static modulus of elasticity of wood is shown in Fig. 2. The relationship between wood static bending (MOR) and dynamic modulus of elasticity of wood (MOE) is shown in Fig. 3. The relationships between wood density and ultrasound wave speed inside the wood are shown in Fig. 4, separately for the tested collections of samples of natural aged wood and contemporary timber. The equations of linear correlations and values of determination coefficients are given on all graphs.

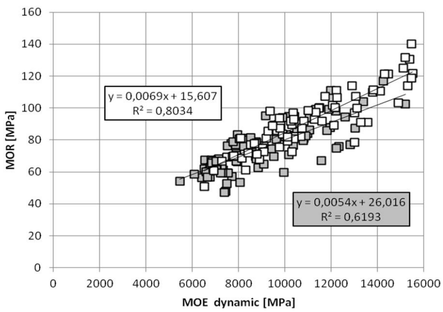

Fig. 3: *The relationship between dynamic MOE and static bending strength (MOR) of natural aged wood (grey points) and contemporary timber (white points).*

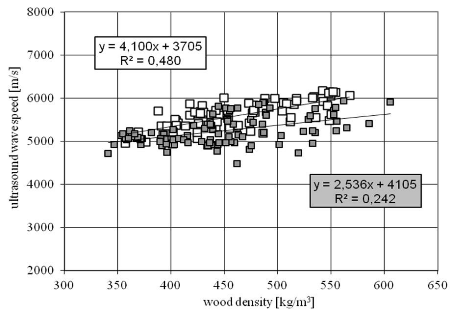

Fig. 4: *The relationship between density and wood transition speed of ultrasounds along the fiber of natural aged wood (grey points) and contemporary timber (white points).*

As it was mentioned above, among laboratory tests on mechanical properties of aged wood from historical constructions, the most common topic is definitely compressive strength parallel to grain (Deppe and Ruhl 1993, Obataya 2007, Witomski et al. 2014, Sonderegger et al. 2015). This probably results from the fact that samples used in such kind of research are of simple shapes and require relatively small amount of original wood from the construction. It is thus rather easy to obtain a significant number of samples for standard tests. This is why the present research on MOR and MOE of natural aged wood was conducted. It seems that in the present research on static bending strength across fibre (MOR), after many years of work a sufficient number of samples of natural aged wood was collected, and compared with contemporary timber, to obtain representative results for Scots pine heartwood. Big difficulty in obtaining natural aged wood being much older than 100 years resulted in many attempts to use accelerated thermo-hydro aged wood to estimate changes in properties of this material (Matejak et al. 1983, Froidevaux et al. 2011, Jankowska and Kozakiewicz 2014).

Most research on properties of natural aged wood has been so far conducted on Japanese wood species (Kohara 1955, Kohara and Okamoto 1956, Hirashima et al. 2005, Ando et al. 2006, Obataya 2007, Yokoyma et al. 2009), among others because of a big number of well preserved old wooden buildings in Japan.

Normal distribution of wood features for both collections of tested samples of natural aged wood and contemporary timber allowed to apply Student test to verify statistical significance of differences between arithmetic averages calculated for both collections.

Average values of MOR, static MOE and dynamic MOE for natural aged wood and fresh cut timber proved to be statistically different. MOR of natural aged wood (74.4 MPa) was visibly lower than MOR of fresh cut timber (91.4 MPa). The obtained results are thus consistent with research conducted on Japanese wood species. Japanese research on wood of *Ch. obtusa* and Z. serrata has proven that natural aged wood more than 300 years old has a lower durability for static bending (MOR) as compared to contemporary timber (Kohara and Okamoto 1956, Obataya 2007). As part of this work on Scots pine wood, the dependence between the age of old wood from individual objects and the decrease in the studied properties has not been captured. The wood was too diverse in density and dating was not precise(no dendrochronological designation).

The transition speed of ultrasounds is a feature related to wood density, yet a more important factor is the anatomical structure of the material. As a result of ageing, gradual changes in the ultrastructure of cell walls (shortening of cellulose chains and increase of crystallinity – e.g. Guo et al. 2018) in the wood appear, which causes a slight reduction of the transition speed of ultrasounds in old wood (Fig. 4.).

Scots pine wood used in historical constructions preserved to this day in Central and Eastern Europe had always low amount of sapwood, or no sapwood at all. If a constructional element contained some sapwood, it was usually strongly damaged by wood boring insects, especially the old house borer *Hylotrupes bajulus* L. Heartwood of Scots pine has a low content of proteins, which does not provide proper conditions for old house borer larvae to survive, which was ascertained many years ago (Becker 1963a, Becker 1963b). This fact results in predominant amount of heartwood in preserved original constructions of historical buildings. Frequently, structural nodes hold on undamaged heartwood. Unfortunately, good quality of original aged wood tends to be underestimated during conservatory work in historical buildings and recognised as poor. This is because the estimation of the condition of roof construction is typically based on visible damages of constructional elements near the surface, where sapwood dominates, showing damage caused by insect larvae. This does not mean, however, that the entire constructional element, consisting mostly of heartwood, is in poor condition. Especially in the case of ceiling beams and rafters, the most important properties seem to be static bending strength across fibre (MOR) and Young's moduli (dynamic MOE and static MOE).

However, natural aged wood is characterised by higher brittleness, which was earlier proven in research on Japanese natural aged wood (Kohara and Okamoto 1956, Hirashima et al. 2005, Obataya 2007, Yokoyma et al. 2009). Aged wood of Scots pine used in present study was of various age, yet it was much younger than the wood from ancient Japanese temples. The wood from rafter in Lidzbark had been cut at the age 130-140 years and since then spent 458 years in the construction. The wood from the church in Cegłów served in the construction for no less than 388 years since the moment it had been cut down. The wood from wall beams in the Church in Puszcza Mariańska came from trees cut down 262 years ago. However, also in the case of the examined Scots pine wood, a decrease was visible in average durability for static bending (MOR) at around 19%. Dealing with wood 250 – 500 years old, such level of decrease in MOR should be taken into account. Also, coefficient of variation of MOR for natural aged wood used in the experiments was 19.4%, thus very close to coefficient of variation of MOR for fresh cut timber (19.7%).

The calculations of wood static bending strength (MOR) and static MOE (Fig. 2) showed a good level of matching of both properties, with the value of $R^2 = 0.843$ for contemporary timber and $R^2 = 0.674$ for natural aged wood. Similarly, in the case of MOR and dynamic MOE, a satisfactory matching was shown by the results ($R^2 = 0.803$ for contemporary timber and $R^2 = 0.619$ for natural aged wood).

In the studied range of wood age (262-458 years since cutting down), the value of MOR is also related to the density of wood (Fig. 1). The coefficient of determination for natural aged Scots pine wood is in this case $R^2 = 0.560$, while for contemporary timber $R^2 = 0.786$. Thus still around 56% of variation in static bending strength (MOR) in aged wood is caused by the influence of wood density.

The decrease in shear strength parallel to grain of natural aged Scots pine wood was insignificant, as was shown by earlier research. Shear strength of natural aged wood was 8.55 MPa, while shear strength of contemporary timber was 8.64 MPa (Krajewski et al. 2016). Compressive strength parallel to grain was much higher for natural aged wood than for contemporary timber (Witomski et al. 2014). This phenomenon had been studied earlier on Japanese wood (Kohara 1954, Kohara and Okamoto 1956, Obataya 2007, Yokoyma et al. 2009).

Average transition speed of ultrasounds along the fibre was higher for natural aged wood than for contemporary timber and the difference was statistically significant, yet the correlation between transition speed of ultrasounds along the fibre and the density of wood was unsatisfactory – below 0.5 (Fig. 4). However, the experiments were conducted on relatively narrow and short samples (30 cm), which acts in favour of noises resulting from reflections of the wave on the edges. Results allowing much better estimation of capabilities of this test method can be achieved on big-dimensional, long wooden elements. In the light of Japanese research, it seems that transition speed of ultrasounds is conditioned not only to wood density, but also to the degree of crystallinity of cellulose. Based on earlier research, it was ascertained that crystallinity of cellulose is higher in natural aged wood (Kohara and Okamoto 1956, Obataya 2007, Nilsson and Rowell 2012, Guo et al. 2018). It is probable that a stronger conditioning of transition speed of ultrasounds along the fibre to crystallinity of cellulose caused a weaker conditioning of the feature to the density of wood. Due to a wide range of the research, experiments conducted so far did not include studies on crystallinity of cellulose and its impact on transition speed of ultrasounds along the fibre of wood. The authors hope to continue their work in this matter.

CONCLUSIONS

The obtained results show that natural aged Scots pine wood, taken from constructions around 250 – 500 years old, is characterised by a wood static bending strength (MOR) lower by almost 1/5 than in the case of contemporary timber. At the same time, MOR of wood is still significantly related to the density of wood.

There is a strong proportional relationship between bending strength and static and dynamic modulus of elasticity in contemporary and old wood. Regardless of the above, the old wood is characterized by significantly lower values of static and dynamic modulus of elasticity along the fibers.

The coefficient of determination R^2 for correlation between transition speed of ultrasounds along the fibre and the density of wood, resulting from tests on relatively small samples, was as low as 0.24 – 0.48 (thus did not exceed 0.5). For this reason, it seems that the estimation of this method of testing static bending strength (MOR), highly correlated with the density of wood (R^2 = 0.86 – 0.97), should be based on tests performed on big-dimensional construction elements and taking crystallinity of cellulose into account.

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